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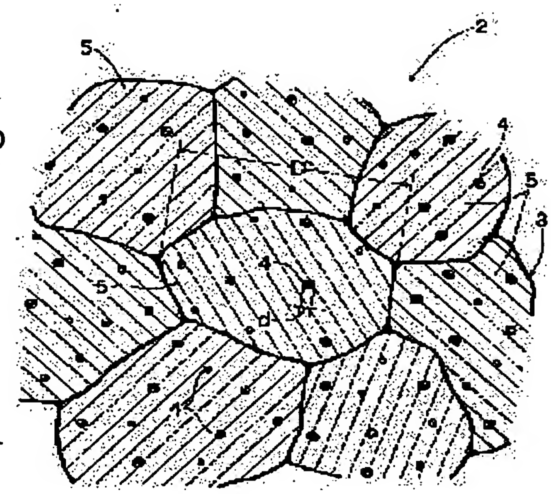
4) HYDROGEN STORAGE ALLOY POWDER AND ON-VEHICLE HYDROGEN STORAGE TANK

7)Abstract:
ꓤOBLEM TO BE SOLVED: To provide hydrogen storage alloy powder ˑge in the quantity of hydrogen to be occluded, further high in hydrogen ;cluding and discharging rate and suitable as the on-vehicle one.
ƆLUTION: Hydrogen storage alloy powder 1 is an aggregate of an Mg atrix 3 and plural ultra-fine particles 4 dispersed into the Mg matrix 3. ıe Mg matrix 3 contains plural Mg crystals 5 having a particle diameter D 1.0 to 500 μm. The particle diameter (d) of the ultra-fine particles is 10 500 nm. The ultra-fine particles 4 are composed of at least one kind ·lected from among Ni ultra-fine particles, Fe ultra-fine particles, V tra-fine particles, Mn ultra-fine particles, Ti ultras-fine particles, Cu tra-fine particles, Al ultra- fine particles, Pd ultra-fine particles, Pt tra-fine particles, Zr ultra-fine particles, Au ultra-fine particles, Ag tra-fine particles, Co ultra-fine particles, Mo ultra-fine particles, Nb tra-fine particles, Cu ultra-fine particles, Zn ultra- fine particles, Ru tra-fine particles, Rh ultra-fine particles, Ta ultra-fine particles, Ir ultra-ıe particles, W ultra-fine particles and their alloy ultra-fine particles.



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laim(s)]
laim 1] Are the aggregate of an alloy particle (2) which has two or more ultrafine particles (4) distributed to Mg itrix (3) and its Mg matrix (3), and the aforementioned Mg matrix (3) two or more Mg crystals (5) whose diameters of crystal grain are 1.0 micrometer<=D <=500micrometer It has and the particle size d of the aforementioned rafine particle (4) is 10 nm<=d<=500nm. the aforementioned ultrafine particle (4) nickel ultrafine particle, nickel oy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, ı ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, . alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy rafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au rafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co oy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine rticle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine particle, Zn alloy ultrafine particle, Ru ultrafine rticle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy rafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle And hydrogen storing metal alloy wder characterized by the thing which is chosen from W alloy ultrafine particle, and which is a kind at least.
laim 2] Hydrogen storing metal alloy powder according to claim 1 whose particle size d of the aforementioned rafine particle (4) is d>=100nm.
laim 3] Content GP of the aforementioned ultrafine particle (4) Hydrogen storing metal alloy powder according to ıim 1 or 2 which is 0.1 atom %<=GP <=5.0 atom %.
laim 4] Content GP of the aforementioned ultrafine particle (4) Hydrogen storing metal alloy powder according to ıim 1 or 2 which is 0.3 atom %<=GP <=3.0 atom %.
laim 5] Two or more aforementioned ultrafine particles (4) are hydrogen storing metal alloy powder according to ıim 1, 2, 3, or 4 which consists of two or more nickel ultrafine particle and two or more Fe ultrafine particles.

:TAILED DESCRIPTION

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etailed Description of the Invention]
)01]
ıe technical field to which invention belongs] this invention relates to the hydrogen storage tank for mount having drogen storing metal alloy powder and its hydrogen storing metal alloy powder.
)02]
escription of the Prior Art] Conventionally, what is depended on mechanical alloying as hydrogen storing metal ɔy powder, the thing pass each process of casting, trituration, and activation are known.
)03]
'oblem(s) to be Solved by the Invention] However, the conventional hydrogen storing metal alloy powder had the ɔblem of being inadequate, about hydrogen storage capacity and hydrogen-absorption discharge speed, in order to ve used it as hydrogen storing metal alloy powder for mount in fuel cell loading vehicles.
)04]
leans for Solving the Problem] While hydrogen storage capacity is size, hydrogen-absorption discharge speed is ick, and this invention aims at offering the aforementioned hydrogen storing metal alloy powder suitable as an object mount.
)05] In order to attain the aforementioned purpose According to this invention, Mg matrix And two or more ultrafine rticles distributed to the Mg matrix It is the aggregate of the alloy particle which it has, and the aforementioned Mg ıtrix has two or more Mg crystals whose diameters D of crystal grain are 1.0 micrometer<=D <=500micrometer, and : particle size d of the aforementioned ultrafine particle is 10 nm<=d<=500nm. the aforementioned ultrafine particle :kel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine rticle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy rafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy rafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr rafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag oy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine rticle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine rticle, The hydrogen storing metal alloy powder which is chosen from Zn alloy ultrafine particle, Ru ultrafine rticle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy rafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle, and W alloy ultrafine particle and ıich is a kind at least is offered.
)06] As mentioned above, hydrogen storing metal alloy powder presents the hydrogen storage capacity as for which iginates in the detailed metal texture, and has very high activity in Mg matrix which has the diameter D of crystal ain of mum order, therefore activation becomes it nothing size since particle size d is the aggregate of an alloy rticle which distributed the ultrafine particle of nm order, and a quick hydrogen-absorption discharge speed.
)07] However, since the aforementioned diameter D of crystal grain becomes complicated [the manufacture process hydrogen storing metal alloy powder] in D< 1.0 micrometers, mass production becomes difficult, and on the other nd by D> 500 micrometers, the hydrogen-absorption discharge speed of hydrogen storing metal alloy powder falls. oreover, in d< 10nm, the activity of an ultrafine particle has the aforementioned, too high particle size d, the handling the particle becomes difficult, and, on the other hand, the hydrogen storage capacity of hydrogen storing metal alloy ıwder serves as smallness by d> 500nm, and hydrogen-absorption discharge speed becomes slow.
)08] According to this invention, moreover, a Ti-Fe alloy matrix And it is the aggregate of an alloy particle which s two or more ultrafine particles distributed to the Ti-Fe alloy matrix. The aforementioned Ti-Fe alloy matrix has two more Ti-Fe alloy crystals whose diameters D of crystal grain are 1.0 micrometer<=D <=500micrometer, and the

ticle size d of the aforementioned ultrafine particle is 10 nm<=d<=500nm. the aforementioned ultrafine particle (4) kel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine ·ticle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy :afine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy :afine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr rafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag ɔy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine ticle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine ticle, The hydrogen storing metal alloy powder which is chosen from Zn alloy ultrafine particle, Ru ultrafine ticle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy rafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle, and W alloy ultrafine particle and ich is a kind at least is offered.

)09] This hydrogen storing metal alloy powder as well as the above presents without activation the hydrogen storage)acity which becomes size, and a quick hydrogen-absorption discharge speed. The reason for limitation of the ırementioned diameter D of crystal grain and the aforementioned particle size d is the same as the aforementioned ;e.

)10] Furthermore, according to this invention, it is a hydrogen storage tank for mount having hydrogen storing metal ɔy powder. the aforementioned hydrogen storing metal alloy powder Mg matrix And two or more ultrafine particles tributed to the Mg matrix It is the aggregate of the alloy particle which it has, and the aforementioned Mg matrix has ɔ or more Mg crystals whose diameters D of crystal grain are 1.0 micrometer<=D <=500micrometer, and the :ticle size d of the aforementioned ultrafine particle is 10 nm<=d<=500nm. the aforementioned ultrafine particle :kel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine :ticle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy rafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy rafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr rafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag oy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine rticle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine rticle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, The hydrogen storage tank for ount which is chosen from Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine rticle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle, and W alloy ultrafine particle and which is :ind at least is offered.

)11] Since this hydrogen storage tank contains the hydrogen storing metal alloy powder which presents the hydrogen ırage capacity which becomes size as mentioned above, and a quick hydrogen-absorption discharge speed, it is .table as an object for mount. The reason for limitation of the aforementioned diameter D of crystal grain and the ɔrementioned particle size d is the same as the aforementioned case.

)12]

mbodiments of the Invention] In drawing 1 and 2, the hydrogen storing metal alloy powder 1 is the aggregate of the oy particle 2, and the alloy particle 2 has two or more ultrafine particles 4 distributed to the Mg matrix 3 and its Mg ıtrix 3. two or more Mg crystals 5 whose diameters D of crystal grain of the Mg matrix 3 are 1.0 micrometer<=D ·500micrometer -- having -- moreover, the particle size d of an ultrafine particle 4 -- 10nm<=d<= -- 500nm is =100nm preferably In this case, let the diameter D of crystal grain of the Mg crystal 5, and particle size d of an rafine particle 4 be the length of those longest portions in a microphotography. This is below the same. Although ly Mg crystals 5 with the aforementioned diameter D of crystal grain are consisted of by the Mg matrix 3, what has e which does not have the aforementioned diameter D of crystal grain outside, or two Mg crystals or more is ntained in it. In the Mg matrix 3, about 50 percent of Mg crystals 5 with the aforementioned diameter D of crystal ain should just exist.

)13] As an ultrafine particle 4 **, nickel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe oy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine rticle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy :rafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag :rafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo oy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, ı ultrafine particle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle,

. alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine rticle, W ultrafine particle And as being chosen from W alloy ultrafine particle, a kind is used as it is few.

)14] As mentioned above, the hydrogen storing metal alloy powder 1 presents the hydrogen storage capacity which ginates in the detailed metal texture, and has very high activity in the Mg matrix 3 which has the diameter D of /stal grain of mum order, therefore becomes it size since particle size d is the aggregate of the alloy particle 2 which stributed the ultrafine particle 4 of nm order, and a quick hydrogen-absorption discharge speed.

)15] It is the content GP of an ultrafine particle 4 that more than hydrogen-storage-capacity 6wt% should be secured. .s set as 0.1 atom %<=GP <=5.0 atom %. This content GP The meaning which uses an ultrafine particle 4 in GP <0.1 ›m % is lost, and, on the other hand, hydrogen storage capacity becomes less than [6wt%] by GP >5.0 atom %. ›ntent GP of an ultrafine particle 4 It is 0.3 atom %<=GP <=3.0 atom % preferably, and, thereby, more than drogen-storage-capacity 7.0wt% can be secured.

)16] As hydrogen storing metal alloy powder 1, the aggregate of the alloy particle 2 which is distributed to the Ti-Fe oy matrix 3 and its Ti-Fe alloy matrix 3 and which has two or more of the same ultrafine particles 4 as the above can mentioned out of the aforementioned thing. in this case, two or more Ti-Fe alloy crystals 5 whose diameters D of /stal grain of the Ti-Fe alloy matrix 3 are 1.0 micrometer<=D <=500micrometer -- having -- moreover, the particle :e d of an ultrafine particle 3 -- 10nm<=d<= -- 500nm is d>=100nm preferably Moreover, content GP of an ultrafine rticle 4 since it is the same as that of the aforementioned thing It is 0.1 atom %<=GP <=5.0 atom %, and is 0.3 atom <=GP <=3.0 atom % preferably. Although it consists of only Ti-Fe alloy crystals 5 which have the aforementioned ımeter D of crystal grain in the Ti-Fe alloy matrix 3, one which does not have the aforementioned diameter of crystal ıin outside, or the thing which has two or more Ti-Fe alloy crystals is also contained. In the Ti-Fe alloy matrix 3, out 50 percent of Ti-Fe alloy crystals 5 with the aforementioned diameter D of crystal grain should just exist.

)17] In manufacture of the hydrogen storing metal alloy powder 1, the method of performing either mechanical oying or mechanical grinding using the ultrafine particle powder which is the aggregate of the Ti-Fe alloy matrix wder which is the aggregate of a Ti-Fe alloy matrix particle, and an ultrafine particle 4, using the ultrafine particle wder which is the aggregate of Mg matrix powder which is the aggregate of Mg matrix particle, and an ultrafine rticle 4 is adopted. In this case, particle size D0 of Mg matrix particle and a Ti-Fe alloy matrix particle 0<=2000 .crometers of 10 micrometer<=D are suitable. Particle size D0 In D0 <10micrometer, since the activity of these ıtrices particle is high, handling nature becomes bad, on the other hand by D0 >2000micrometer, the hydrogen ›rage capacity of hydrogen storing metal alloy powder serves as smallness, and hydrogen-absorption discharge speed comes slow.

)18] Hereafter, an example is explained.

)19] [Example 1] Purity is 99.9% and it is particle size D0. With the aggregate of Mg matrix particle which is D0 :500micrometer, purity is 99.9%, particle size d is [the aggregate of 20 nm<=d<=400nm (200nm of mean particle ımeters) nickel ultrafine particle and purity] 99.9%, and particle size d prepared the aggregate of 100 ı<=d<=500nm (300nm of mean particle diameters) Fe ultrafine particle. And weighing capacity of these particles ıs carried out so that the sum of the content of the aggregate of nickel ultrafine particle and the aggregate of Fe rafine particle to the aggregate of Mg matrix particle might change within the limits of 0.05 atom % to 10.0 atom % d it might be set to the interior division injury of the sum of each content, and nickel ultrafine particle:Fe ultrafine rticle **2:1, and the mixed-powder end of 15 sorts which it is 100g in total, and differs in a compounding ratio was tained.

020] Each end of mixed powder is put into a pot (product made from JIS SUS316) with a capacity [of a horizontal-pe ball mill (product made from Honda)] of 2500ml with 990 balls (product made from JIS SUS316) with a ımeter of 10mm, the inside of a pot is held in the hydrogen gas atmosphere of 1.0MPa(s), and it is a pot rotational equency. 64rpm, milling time t Ball milling was performed on the conditions of 4 hours. In this case, in a pot, it is avitational acceleration GP. Acceleration 0.2G 0.2 times as many as this had occurred. Hydrogen storing metal alloy ›wder was extracted in the atmosphere after ball milling.

021] Since it was hydrogenated in the ball milling process, each hydrogen storing metal alloy powder performed hydrogenation processing which performs vacuum length to them on 350 degrees C and the conditions of 1 hour, d, subsequently carried out the hydrogen-absorption examination which performs high-pressure hydrogen essurization of 1.1MPa(s) from a vacua at the measurement temperature of 310 degrees C about each hydrogen pring metal alloy powder.

022] Table 1 is the sum GP of the content of the diameter D of crystal grain of Mg crystal in Mg matrix about camples 1-15 of each hydrogen storing metal alloy powder, nickel ultrafine particle, and Fe ultrafine particle ickel+Fe and the remainder are Mg), i.e., the content of an ultrafine particle. It is the content GP to a row. An inner vision (nickel, Fe) and hydrogen storage capacity SH It is shown.

23]
ble 1]

素吸合金末	Mgマトリックスにおける Mg結晶の結晶粒径 D (μm)	Ni, Fe超微粒子の含有量 G_P (原子%)			水素吸蔵量 S_H (wt%)
		Ni+Fe	Ni	Fe	
刂1	500≦ ≦800	0.05	0.033	0.017	5.4
刂2	200≦ ≦450	0.10	0.067	0.033	6.8
刂3	50≦ ≦400	0.20	0.134	0.066	6.8
刂4	5≦ ≦400	0.30	0.200	0.100	7.4
刂5	1.5≦ ≦280	0.50	0.333	0.167	7.4
刂6	3≦ ≦350	0.70	0.467	0.233	7.4
刂7	2≦ ≦330	1.00	0.667	0.333	7.3
刂8	1.1≦ ≦350	1.50	1.000	0.500	7.2
刂9	4≦ ≦390	2.00	1.333	0.667	7.2
10	3≦ ≦400	3.00	2.000	1.000	7.0
11	3.5≦ ≦410	4.00	2.667	1.333	6.4
12	2.5≦ ≦300	5.00	3.333	1.667	6.2
13	1.5≦ ≦320	6.00	4.000	2.000	5.3
14	1.9≦ ≦250	8.00	5.333	2.667	5.0
15	2≦ ≦100	10.00	6.667	3.333	4.9

)24] Drawing 3 is based on Table 1 and is the sum (nickel+Fe) GP of the content of nickel ultrafine particle and Fe rafine particle. Hydrogen storage capacity SH A relation is graph-ized. So that clearly from Table 1 and drawing 3 in : case of Examples 2-12 It is in within the limits whose diameter D of crystal grain of Mg crystal in Mg matrix is 1.0 crometer<=D <=500micrometer. Moreover, the sum of the aforementioned content (nickel+Fe), that is, content GP /drogen storage capacity SH since it is set as 0.10 atom %<=GP <=5.0 atom % it raises to SH >=6wt% -- having -- ** -- especially -- like Examples 4-10 -- the amount GP of contents if it is set as 0.3 atom %<=GP <=3.0 atom % -- drogen storage capacity SH It is possible to raise further, as it was called 7.0wt%<=SH <=7.4wt%.
)25] Next, PCT measurement was performed about Examples 5 and 10 after the aforementioned dehydrogenation ocessing. Drawing 4 is Example 5 and drawing 5 shows the PCT property (convergence time :; during 5 minutes 310 grees C, occlusion discharge) of Example 10, respectively. Hydrogen storage capacity SH of drawing 4 and 5 to the amples 5 and 10 As it was called 7.0wt% 7.4wt(s)%, respectively, it is size, and hydrogen-absorption discharge eed is very quick, and it turns out that the highest hydrogen-absorption burst size is presented in the convergence ne for 5 minutes.
026] [Example 2] Purity is 99.9% and it is particle size D0. With the aggregate of the Ti50Fe50 alloy matrix particle numerical unit is atomic %) which is D0 <=300micrometer, purity is 99.9% and particle size d prepared the gregate of 100 nm<=d<=500nm (300nm of mean particle diameters) Fe ultrafine particle. And content GP of the gregate of Fe ultrafine particle to the aggregate of a Ti50Fe50 alloy matrix particle Weighing capacity was carried it so that it might become GP =1 atom %, and the mixed-powder end of a total of 25g was obtained. This end of

xed powder is put into a pot (product made from JIS SUS316) with a capacity [of a horizontal-type ball mill oduct made from Honda)] of 800ml with 180 balls (product made from JIS SUS316) with a diameter of 10mm, the ide of a pot is held in the hydrogen gas atmosphere of 1.0MPa(s), and it is a pot rotational frequency. 85rpm, milling ıe t Ball milling was performed on the conditions of 1 hour. In this case, in the pot, acceleration 0.1G 0.1 times as ıch as gravitational acceleration G had occurred. Hydrogen storing metal alloy powder was extracted in the nosphere after ball milling. The diameter D of crystal grain of the Ti50Fe50 alloy crystal in the Ti50Fe50 alloy ıtrix of this hydrogen storing metal alloy powder was 1.5 micometer<=D <=450micrometer. Let this end of an alloy wder be Example 16.

)27] Dehydrogenation processing was performed to Example 16 on the same conditions as an example 1, ›sequently, Example 16 was held under the hydrogen pressure force of 1.1MPa(s) using PCT equipment, and change the hydrogen pressure force accompanying time progress was measured. <: [A HREF="/Tokujitu/tjitemdrw.ipdl? ı000=237&N0500=1E_N/;>;?] <6=9///&N0001=438&N0552=9&N0553=000009" TARGET="tjitemdrw"> drawing ·hows a measurement result, and Example 17 is the same composition as Example 16, and is equivalent to the nventional example by casting. Although the hydrogen pressure force is low in Example 16 so that clearly from ıwing 6, it turns out that the occlusion of hydrogen is appearing simultaneously with hydrogen introduction. Since : hydrogen pressure force is low while Example 17 is subvital, the occlusion of hydrogen does not appear after 300-ur progress.

)28] [Example 3] Drawing 7 and hydrogen storage tank T for mount shown in 8 have the pressure-resistant outer ;e object 6 of the cross-section round shape which consisted of stainless steel etc., and the inside of the outer case ject 6 is divided with the permeability filter 7 made from nickel by the small space 9 by the side of the end wall 8, d 11 between the skies by the side of the other end wall 10. The permeability filter 7 has the micropore of a large mber which hydrogen can frequent, for example, the hole which is several nm - 0.5 micrometers. As a component of : permeability filter 7, metallic materials, such as stainless steel, ceramics, etc. are used out of nickel.

)29] It fills up with the hydrogen storing metal alloy powder 1 in 11 between the skies, and the flow conduit 12 of : hydrogen for occlusion and discharge hydrogen penetrates, and is held at the end wall 8 by the side of the small ace 9.

)30] The shell 13 which consists of stainless steel etc. made the axis agree in the axis of the outer case object 6, and s penetrated the outer case object 6, the permeability filter 7, and the hydrogen storing metal alloy powder 1, and the ıl of the penetration section of the shell 13 in the ends walls 8 and 10 and the permeability filter 7 is carried out. The ;ide of a shell 13 is used as a path which circulates the fluid for cooling at the time of hydrogen absorption, and is ed as a path which circulates a heating fluid at the time of hydrogen desorption.

)31] The aforementioned powder is used as hydrogen storing metal alloy powder 1. That is, the powder is the gregate of the alloy particle 2 which has two or more ultrafine particles 4 distributed to the Mg matrix 3 and its Mg ıtrix 3. The Mg matrix 3 has two or more Mg crystals whose diameters D of crystal grain are 1.0 micrometer<=D ·500micrometer, and the particle size d of an ultrafine particle 4 is 10 nm<=d<=500nm. Those ultrafine particles 4 :kel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine rticle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy rafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy rafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr rafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag oy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine rticle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine rticle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy rafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W rafine particle And it is it a kind that it is few as being chosen from W alloy ultrafine particle.

032] Since this hydrogen storage tank T builds in the hydrogen storing metal alloy powder 1 which presents the drogen storage capacity which becomes size as mentioned above, and a quick hydrogen-absorption discharge speed, is suitable as an object for mount.

033]

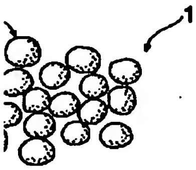
ffect of the Invention] according to invention according to claim 1 to 9, hydrogen storage capacity is size by nstituting as mentioned above -- both hydrogen-absorption discharge speed is quick, and hydrogen storing metal oy powder suitable as an object for mount can be offered

034] According to invention according to claim 10, a hydrogen storage tank suitable as an object for mount can be fered.

IOTICES *

:AWINGS

:awing 1]



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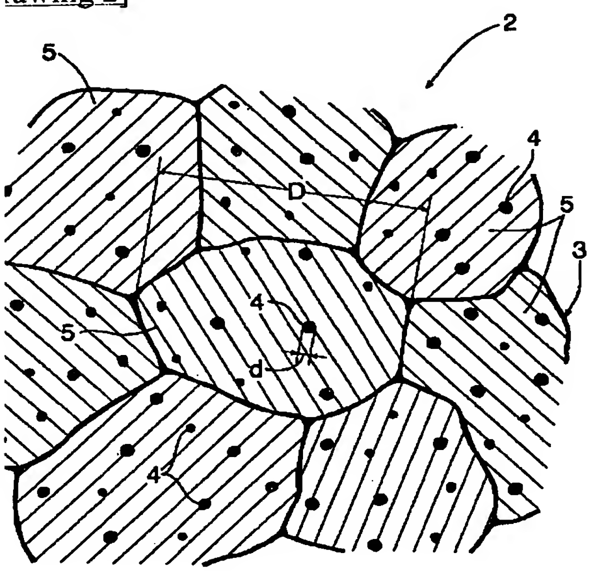

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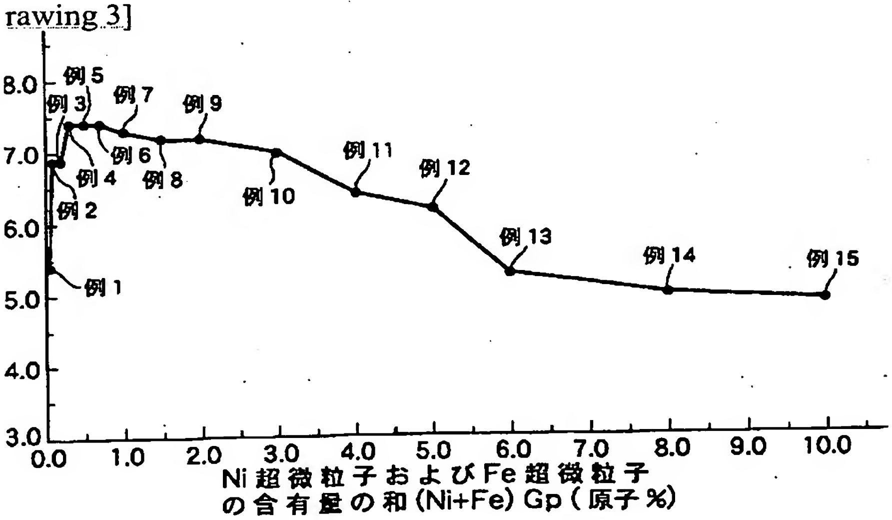

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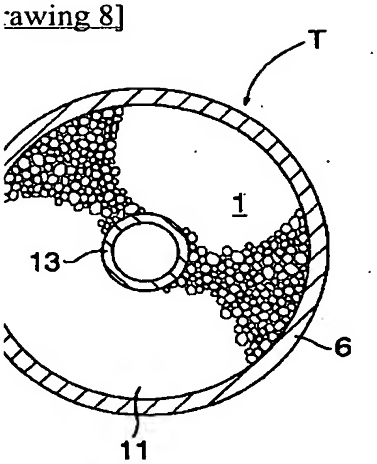

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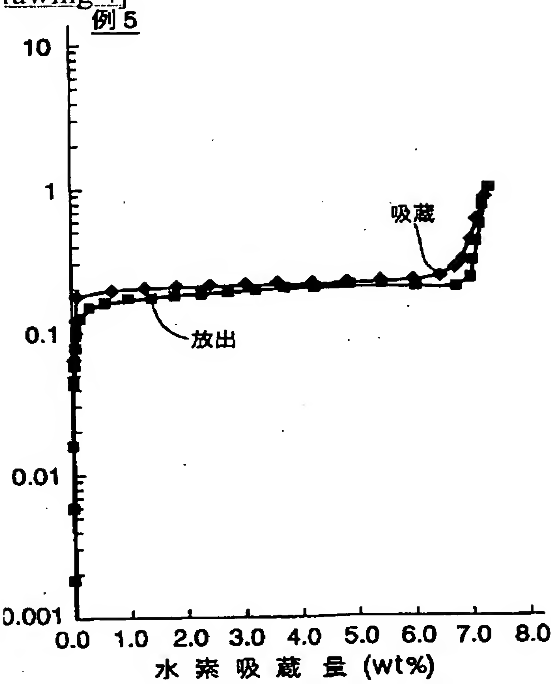

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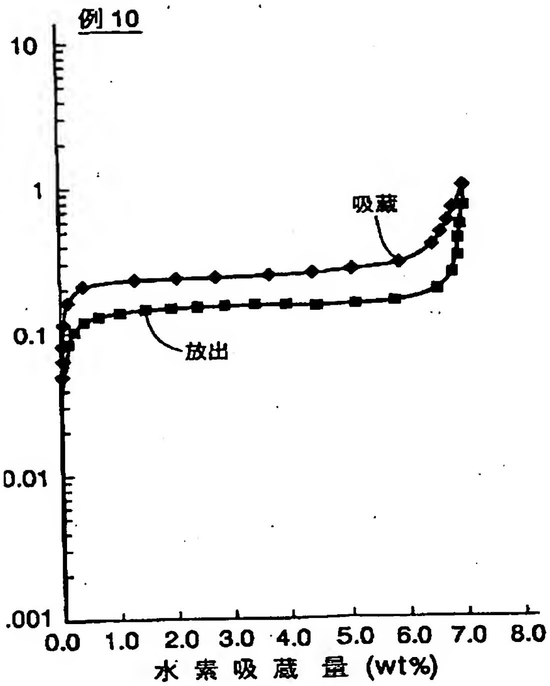

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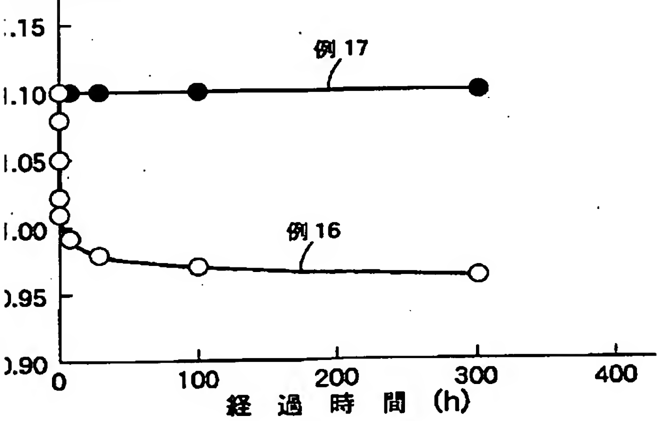

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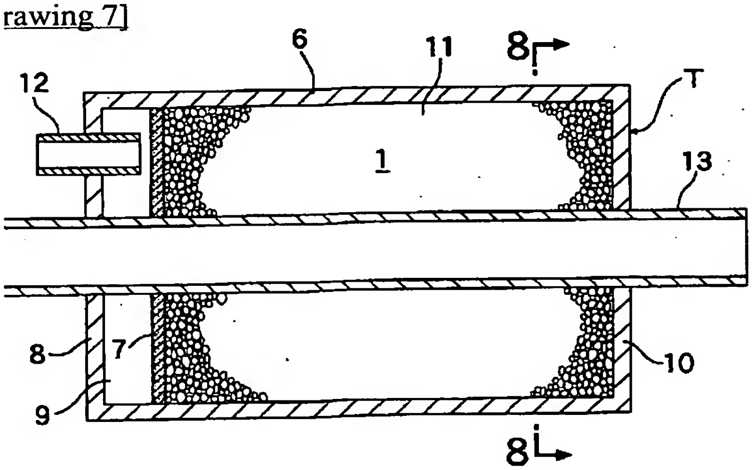

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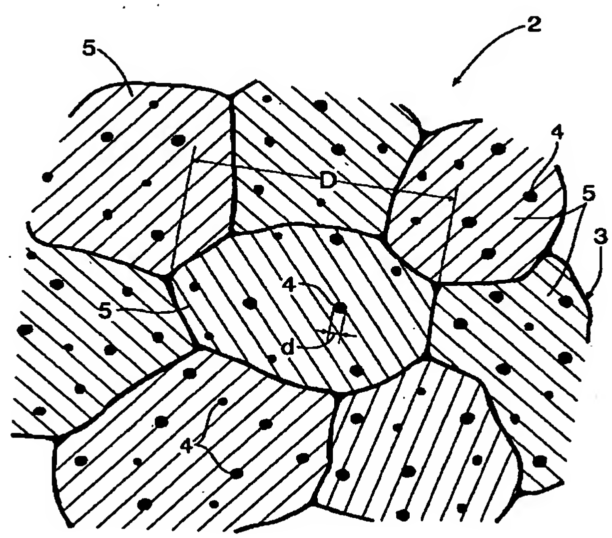
(54)【発明の名称】　水素吸蔵合金粉末および車載用水素貯蔵タンク

(57)【要約】
【課題】　水素吸蔵量が大であると共に水素吸蔵放出速度が速く，車載用として好適な水素吸蔵合金粉末を提供する。
【解決手段】　水素吸蔵合金粉末１は，Ｍｇマトリックス３およびそのＭｇマトリックス３に分散する複数の超微粒子４を有する合金粒子２の集合体である。Ｍｇマトリックス３は結晶粒径Ｄが１．０μｍ≦Ｄ≦５００μｍである複数のＭｇ結晶５を有する。超微粒子４の粒径ｄは１０ｎｍ≦ｄ≦５００ｎｍである。超微粒子４は，Ｎｉ超微粒子，Ｆｅ超微粒子，Ｖ超微粒子，Ｍｎ超微粒子，Ｔｉ超微粒子，Ｃｕ超微粒子，Ａｌ超微粒子，Ｐｄ超微粒子，Ｐｔ超微粒子，Ｚｒ超微粒子，Ａｕ超微粒子，Ａｇ超微粒子，Ｃｏ超微粒子，Ｍｏ超微粒子，Ｎｂ超微粒子，Ｃｒ超微粒子，Ｚｎ超微粒子，Ｒｕ超微粒子，Ｒｈ超微粒子，Ｔａ超微粒子，Ｉｒ超微粒子，W超微粒子およびそれらの合金超微粒子から選択される少なくとも一種である。



水素吸蔵合金粉末は，それを燃料電池搭載車両における車載用水素吸蔵合金粉末として用いるには，水素吸蔵量および水素吸蔵放出速度に関し不十分である，という問題があった。

【０００４】

【課題を解決するための手段】本発明は，水素吸蔵量が大であると共に水素吸蔵放出速度が速く，車載用として好適な前記水素吸蔵合金粉末を提供することを目的とする。

【０００５】前記目的を達成するため本発明によれば，Ｍｇマトリックスおよびそのマトリックスに分散する複数の超微粒子を有する合金粒子の集合体であって，前記Ｍｇマトリックスは結晶粒径Ｄが１．０μｍ≦Ｄ≦５００μｍである複数のＭｇ結晶を有し，また前記超微粒子の粒径ｄは１０ｎｍ≦ｄ≦５００ｎｍであり，前記超微粒子は，Ｎｉ超微粒子，Ｎｉ合金超微粒子，Ｆｅ超微粒子，Ｆｅ合金超微粒子，Ｖ超微粒子，Ｖ合金超微粒子，Ｍｎ超微粒子，Ｍｎ合金超微粒子，Ｔｉ超微粒子，Ｔｉ合金超微粒子，Ｃｕ超微粒子，Ｃｕ合金超微粒子，Ａｌ超微粒子，Ａｌ合金超微粒子，Ｐｄ超微粒子，Ｐｄ合金超微粒子，Ｐｔ超微粒子，Ｐｔ合金超微粒子，Ｚｒ超微粒子，Ｚｒ合金超微粒子，Ａｕ超微粒子，Ａｕ合金超微粒子，Ａｇ超微粒子，Ａｇ合金超微粒子，Ｃｏ超微粒子，Ｃｏ合金超微粒子，Ｍｏ超微粒子，Ｍｏ合金超微粒子，Ｎｂ超微粒子，Ｎｂ合金超微粒子，Ｃｒ超微粒子，Ｃｒ合金超微粒子，Ｚｎ超微粒子，Ｚｎ合金超微粒子，Ｒｕ超微粒子，Ｒｕ合金超微粒子，Ｒｈ超微粒子，Ｒｈ合金超微粒子，Ｔａ超微粒子，Ｔａ合金超微粒子，Ｉｒ超微粒子，Ｉｒ合金超微粒子，Ｗ超微粒子およびＷ合金超微粒子から選択される少なくとも一種である水素吸蔵合金粉末が提供される。

【０００６】前記のように，水素吸蔵合金粉末は，μｍオーダの結晶粒径Ｄを有するＭｇマトリックスに，粒径ｄがｎｍオーダの超微粒子を分散させた合金粒子の集合体であることから，その微細金属組織に起因して非常に高い活性を有し，したがって，活性化処理無しに，大なる水素吸蔵量と速い水素吸蔵放出速度を呈する。

【０００７】ただし，前記結晶粒径ＤがＤ＜１．０μｍでは水素吸蔵合金粉末の製造過程が複雑となるため量産が困難となり，一方，Ｄ＞５００μｍでは水素吸蔵合金粉末の水素吸蔵放出速度が低下する。また前記粒径ｄがｄ＜１０ｎｍでは超微粒子の活性が高すぎて，その粒子の取扱いが難しくなり，一方，ｄ＞５００ｎｍでは水素吸蔵合金粉末の水素吸蔵量が小となり，また水素吸蔵放出速度が遅くなる。

【０００８】また本発明によれば，Ｔｉ－Ｆｅ合金マトリックスおよびそのＴｉ－Ｆｅ合金マトリックスに分散する複数の超微粒子を有する合金粒子の集合体であって，前記Ｔｉ－Ｆｅ合金マトリックスは結晶粒径Ｄが１．０μｍ≦Ｄ≦５００μｍである複数のＴｉ－Ｆｅ合金結晶を有し，また前記超微粒子の粒径ｄは１０ｎｍ≦ｄ≦５００ｎｍであり，前記超微粒子（４）は，Ｎｉ超微粒子，Ｎｉ合金超微粒子，Ｆｅ超微粒子，Ｆｅ合金超微粒子，Ｖ超微粒子，Ｖ合金超微粒子，Ｍｎ超微粒子，Ｍｎ合金超微粒子，Ｔｉ超微粒子，Ｔｉ合金超微粒子，Ｃｕ超微粒子，Ｃｕ合金超微粒子，Ａｌ超微粒子，Ａｌ合金超微粒子，Ｐｄ超微粒子，Ｐｄ合金超微粒子，Ｐｔ超微粒子，Ｐｔ合金超微粒子，Ｚｒ超微粒子，Ｚｒ合金超微粒子，Ａｕ超微粒子，Ａｕ合金超微粒子，Ａｇ超微粒子，Ａｇ合金超微粒子，Ｃｏ超微粒子，Ｃｏ合金超微粒子，Ｍｏ超微粒子，Ｍｏ合金超微粒子，Ｎｂ超微粒子，Ｎｂ合金超微粒子，Ｃｒ超微粒子，Ｃｒ合金超微粒子，Ｚｎ超微粒子，Ｚｎ合金超微粒子，Ｒｕ超微粒子，Ｒｕ合金超微粒子，Ｒｈ超微粒子，Ｒｈ合金超微粒子，Ｔａ超微粒子，Ｔａ合金超微粒子，Ｉｒ超微粒子，Ｉｒ合金超微粒子，Ｗ超微粒子およびＷ合金超微粒子から選択される少なくとも一種である水素吸蔵合金粉末が提供される。

【０００９】この水素吸蔵合金粉末も前記同様に，活性化処理無しに，大なる水素吸蔵量と速い水素吸蔵放出速度を呈する。前記結晶粒径Ｄおよび前記粒径ｄの限定理由は前記の場合と同じである。

【００１０】さらに本発明によれば，水素吸蔵合金粉末を内蔵した車載用水素貯蔵タンクであって，前記水素吸蔵合金粉末は，ＭｇマトリックスおよびそのＭｇマトリックスに分散する複数の超微粒子を有する合金粒子の集合体であり，前記Ｍｇマトリックスは結晶粒径Ｄが１．０μｍ≦Ｄ≦５００μｍである複数のＭｇ結晶を有し，また前記超微粒子の粒径ｄは１０ｎｍ≦ｄ≦５００ｎｍであり，前記超微粒子は，Ｎｉ超微粒子，Ｎｉ合金超微粒子，Ｆｅ超微粒子，Ｆｅ合金超微粒子，Ｖ超微粒子，Ｖ合金超微粒子，Ｍｎ超微粒子，Ｍｎ合金超微粒子，Ｔｉ超微粒子，Ｔｉ合金超微粒子，Ｃｕ超微粒子，Ｃｕ合金超微粒子，Ａｌ超微粒子，Ａｌ合金超微粒子，Ｐｄ超微粒子，Ｐｄ合金超微粒子，Ｐｔ超微粒子，Ｐｔ合金超微粒子，Ｚｒ超微粒子，Ｚｒ合金超微粒子，Ａｕ超微粒子，Ａｕ合金超微粒子，Ａｇ超微粒子，Ａｇ合金超微粒子，Ｃｏ超微粒子，Ｃｏ合金超微粒子，Ｍｏ超微粒子，Ｍｏ合金超微粒子，Ｎｂ超微粒子，Ｎｂ合金超微粒子，Ｃｒ超微粒子，Ｃｒ合金超微粒子，Ｚｎ超微粒子，Ｚｎ合金超微粒子，Ｒｕ超微粒子，Ｒｕ合金超微粒子，Ｒｈ超微粒子，Ｒｈ合金超微粒子，Ｔａ超微粒子，Ｔａ合金超微粒子，Ｉｒ超微粒子，Ｉｒ合金超微粒子，Ｗ超微粒子およびＷ合金超微粒子から選択される少なくとも一種である車載用水素貯蔵タンクが提供される。

【００１１】この水素貯蔵タンクは，前記のように大なる水素吸蔵量と速い水素吸蔵放出速度を呈する水素吸蔵合金粉末を内蔵しているので，車載用として好適である。前記結晶粒径Ｄおよび前記粒径ｄの限定理由は前記の場合と同じである。

【００２１】各水素吸蔵合金粉末は，そのボールミリング過程において水素化されているので，それらに，３５０℃，１時間の条件で真空引きを行う脱水素化処理を施し，次いで，各水素吸蔵合金粉末について，測定温度３１０℃にて真空状態から１．１ＭＰａの高圧水素加圧を行う水素吸蔵試験を実施した。

【００２２】表１は，各水素吸蔵合金粉末の例１～１５に関するＭｇマトリックスにおけるＭｇ結晶の結晶粒径Ｄ，Ｎｉ超微粒子およびＦｅ超微粒子の含有量の和（Ｎｉ＋Ｆｅ，残部はＭｇ），つまり超微粒子の含有量G_Pならびにその含有量G_Pの内分け（Ｎｉ，Ｆｅ）と，水素吸蔵量S_Hを示す。

【００２３】

【表１】

水素吸蔵合金粉末	ＭｇマトリックスにおけるＭｇ結晶の結晶粒径Ｄ（μｍ）	Ｎｉ，Ｆｅ超微粒子の含有量G_P（原子％）			水素吸蔵量S_H（wt%）
		Ni＋Fe	Ｎｉ	Ｆｅ	
例１	５００≦ ≦８００	0.０５	0.０３３	0.０１７	5.４
例２	２００≦ ≦４５０	0.１０	0.０６７	0.０３３	6.８
例３	５０≦ ≦４００	0.２０	0.１３４	0.０６６	6.８
例４	５≦ ≦４００	0.３０	0.２００	0.１００	7.４
例５	1.５≦ ≦２８０	0.５０	0.３３３	0.１６７	7.４
例６	３≦ ≦３５０	0.７０	0.４６７	0.２３３	7.４
例７	２≦ ≦３３０	1.００	0.６６７	0.３３３	7.３
例８	1.１≦ ≦３５０	1.５０	1.０００	0.５００	7.２
例９	４≦ ≦３９０	2.００	1.３３３	0.６６７	7.２
例１０	３≦ ≦４００	3.００	2.０００	1.０００	7.０
例１１	3.５≦ ≦４１０	4.００	2.６６７	1.３３３	6.４
例１２	2.５≦ ≦３００	5.００	3.３３３	1.６６７	6.２
例１３	1.５≦ ≦３２０	6.００	4.０００	2.０００	5.３
例１４	1.９≦ ≦２５０	8.００	5.３３３	2.６６７	5.０
例１５	２≦ ≦１００	１0.００	6.６６７	3.３３３	4.９

【００２４】図３は表１に基づいてＮｉ超微粒子およびＦｅ超微粒子の含有量の和（Ｎｉ＋Ｆｅ）G_Pと水素吸蔵量S_Hとの関係をグラフ化したものである。表１，図３から明らかなように，例２～１２の場合は，Ｍｇマトリックスにおけるｍｇ結晶の結晶粒径Ｄが１．０μｍ≦Ｄ≦５００μｍの範囲内にあり，また前記含有量の和（Ｎｉ＋Ｆｅ），つまり，含有量G_Pが０．１０原子％≦G_P≦５．０原子％に設定されていることから，水素吸蔵量S_HはS_H≧６ｗｔ％に高められており，特に，例４～１０の如く，含量量G_Pを０．３原子％≦G_P≦３．０原子％に設定すると，水素吸蔵量S_Hを７．０ｗｔ％≦S_H≦７．４ｗｔ％といったように一層高めることが可能である。

【００２５】次に，前記脱水素化処理後の例５，１０についてＰＣＴ測定を行った。図４は例５の，また図５は例１０のＰＣＴ特性（収束時間：５分間；３１０℃，吸蔵放出）をそれぞれ示す。図４，５から，例５，１０の水素吸蔵量S_Hがそれぞれ７．４ｗｔ％，７．０ｗｔ％といったように大であり，また水素吸蔵放出速度が非常に速く，５分間の収束時間において最高水素吸蔵放出量を呈することが判る。

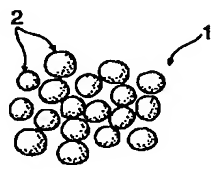
【００２６】〔実施例２〕純度が９９．９％であり，且

３………Ｍｇマトリックス，Ｔｉ－Ｆｅ合金マトリックス

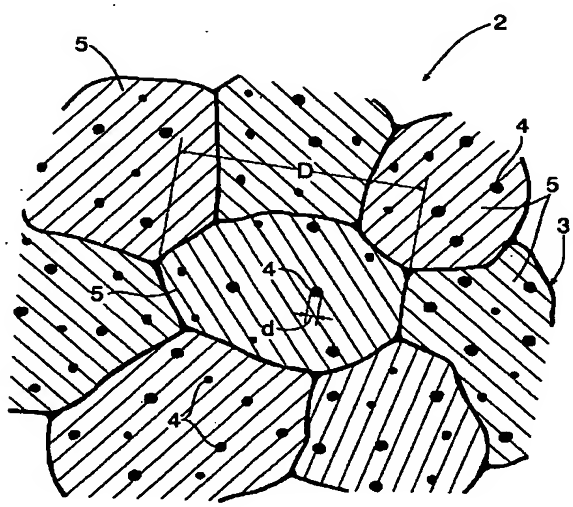
４………超微粒子

Ｔ………水素貯蔵タンク

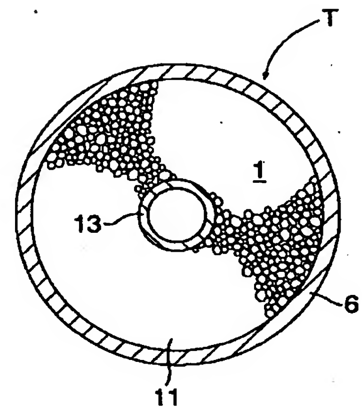
【図１】



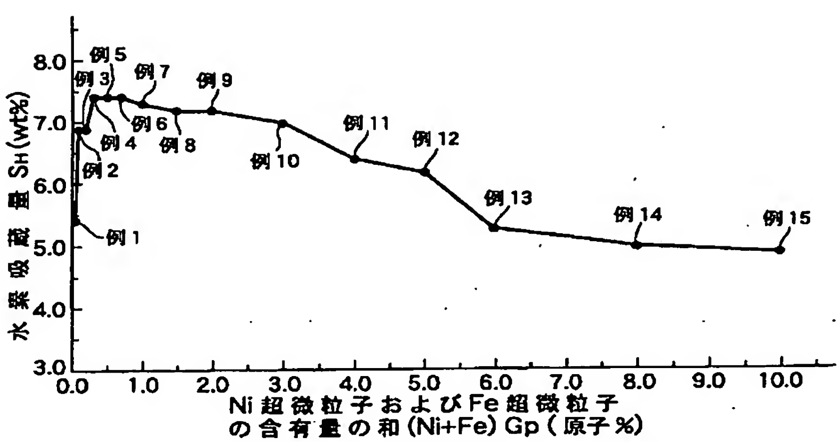
【図２】



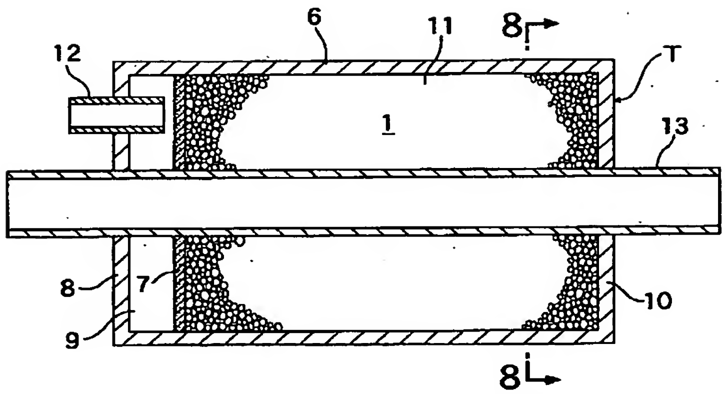
【図８】



【図３】



【図７】



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